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TECHNICAL REPORT 26

Contract N9onr-85801

Project Designation NR031-364

COLD TRAPPING EFFECTS OF A FREEZE
SEAL ON A SODIUM FILLED NICKEL SYSTEM
and
SOME EFFECTS OF A NICKEL COLD TRAP ON
A SODIUM-FILLED STAINLESS STEEL SYSTEM

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By direction of _____

Chief of Naval Research (Code **429**)

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MINE SAFETY APPLIANCES COMPANY

Calhery, Pennsylvania

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C O N F I D E N T I A L

TECHNICAL REPORT 26

Contract N9onr-85801
Project Designation NR031-364

Part I

COLD TRAPPING EFFECTS OF A FREEZE SEAL
IN THE DRAIN-FILL PIPE ON A SODIUM-FILLED NICKEL SYSTEM

Part II

SOME EFFECTS OF A NICKEL COLD TRAP ON A
SODIUM-FILLED STAINLESS STEEL SYSTEM

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File No. 689

Mine Safety Appliances Company
Callery, Pa.
January 4, 1954

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ABSTRACTS

Part I. An all nickel system, containing sodium, was used for iron corrosion studies. After shutdown the drain leg which operated as a freeze seal was analyzed for sodium, sodium monoxide and iron. A maximum bulk density of 60 wt. % Na_2O was found in a section 1 1/2 in. long, 1 1/4 in. from the flowing stream: the bulk density dropped with increasing distance from the stream. Of the 205 mg. of iron added to the system only about 1.5 mg. was found in the cold drain leg.

Part II. Small amounts of iron were moved to a nickel cold trap from a sodium-filled stainless steel system at 1000°F and <0.005 wt. % oxygen concentration. A total of 0.22 grams of iron was found after four months operation with the bottom of the cold trap at 170 - 200°F.

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Part I

Cold Trapping Effects of a Solid Freeze Seal on a Drain-Fill Pipe of a Sodium Filled Nickel System

AUTHORIZATION

This project was authorized by the task order of Contract N9onr-85801.

SUMMARY

This report describes an analytical study of the cold trapping effects of a freeze seal in the drain and fill pipe of a sodium system. An occasion arose wherein the drain-fill pipe of a loop of all nickel construction used for iron and oxygen solubility studies could be sectioned and the contents analyzed for sodium, sodium oxide and iron. The highest oxide concentration found was 0.73 gm $\text{Na}_2\text{O}/\text{cc}$ of pipe volume in the first 1 1/2 in. long section which was 1 1/4 in. from the flowing stream compared with 0.0017 gm $\text{Na}_2\text{O}/\text{cc}$ of pipe volume found in a 1 1/2 in. long section 13 inches from the flowing stream. An iron sample had been exposed to the sodium stream and less than 1% of the 205 mg of iron added to the system was found in the drain pipe contents.

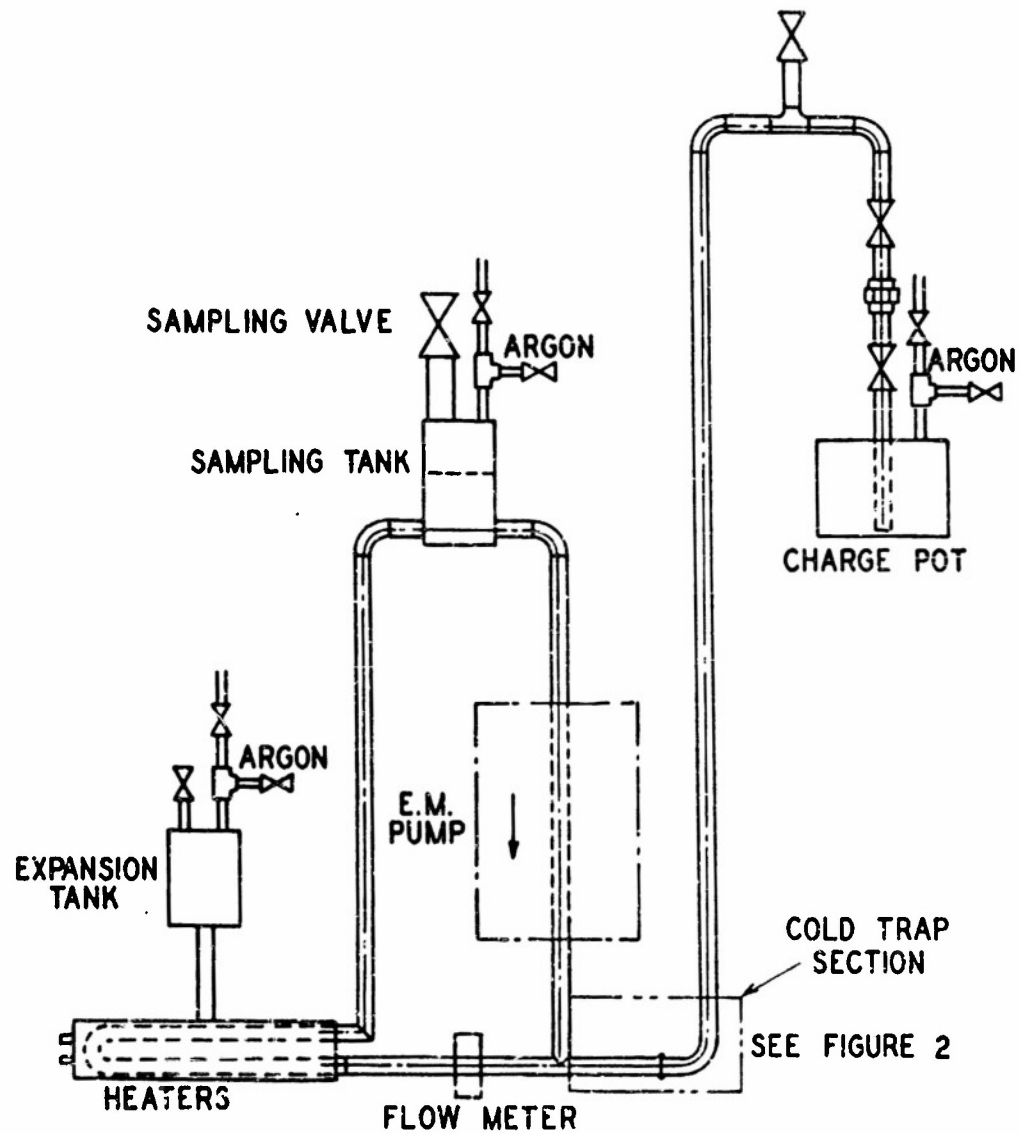
INTRODUCTION

The drain pipe was connected to the loop as shown in Figure 1. Before beginning the series of experiments during which oxide collected in the pipe, the loop had been charged and drained several times without any difficulty. Prior to removal of the drain pipe, the loop had been in operation for four months with the sodium at 1000°F, during which time the oxygen concentration in the stream had been raised as high as 0.050 wt. % by adding Na_2O_2 . During this time of operation iron was exposed to the sodium at various oxygen concentrations by suspending samples of Armco iron in the sampling tank.

TECHNIQUE OF ANALYSIS

The loop was not drained but with the pump off the sodium was simply allowed to freeze in the system. The drain leg was then cut 3/4 in. from the flowing stream. A filter flask with argon flowing through the side-arm was placed over the open end of the cut. A section of the pipe about 1/2 in. long was then cut off and discarded because the first cut had been exposed to the atmosphere. By careful manipulation in cutting nearly through the wall and then breaking the section off inside the flask it was possible to keep contamination of the sections at a low level. There probably was oxide

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ALL NICKEL LOOP

PART I FIGURE 1

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contamination during sectioning and transfer but the last pipe section analyzed indicated that this contamination was low enough to be disregarded.

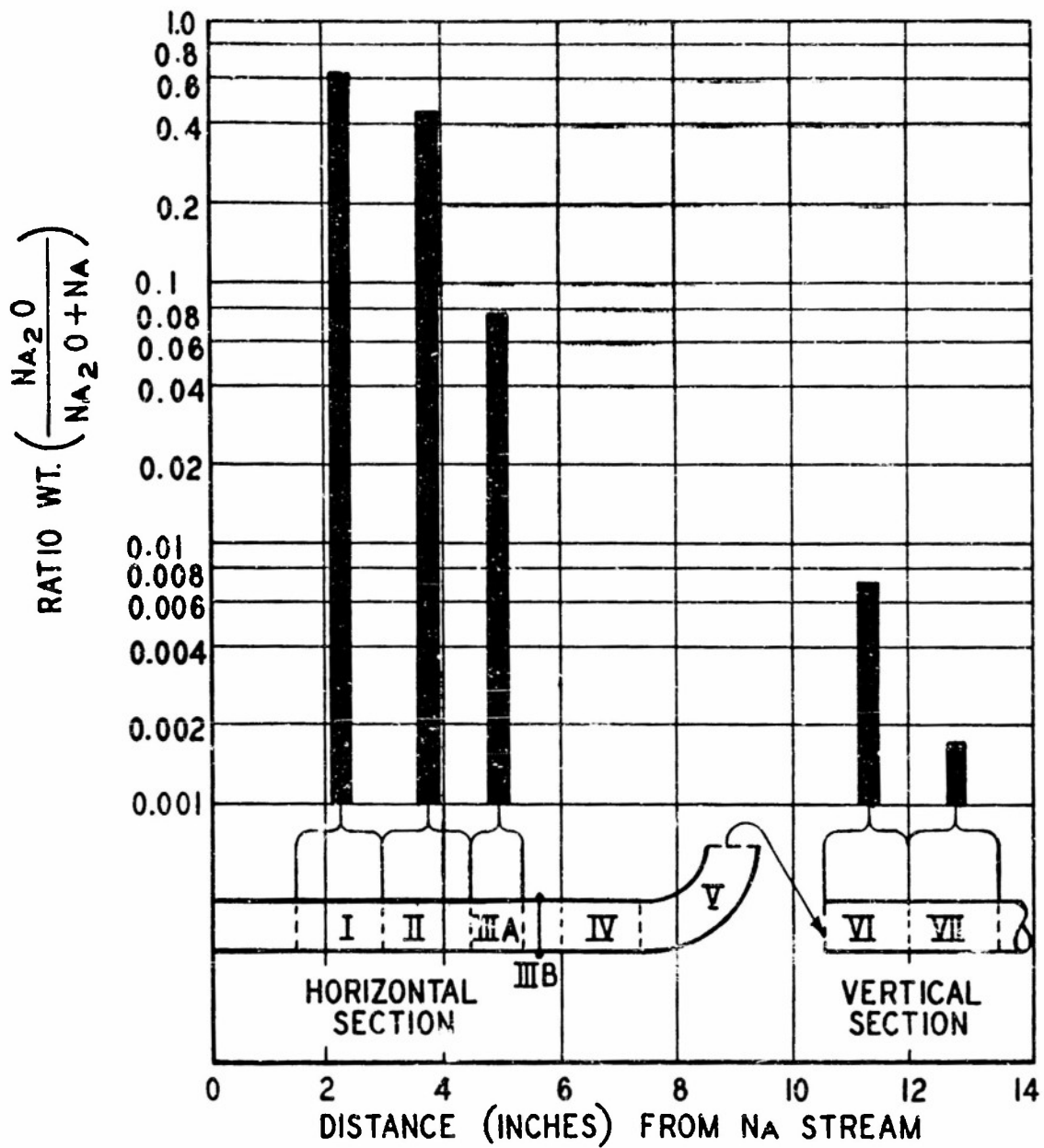
Each section was amalgamated in a chamber such as that used for oxygen-in-sodium analyses. The oxide present in the sections was quite evident, particularly in the sections containing high concentrations. The crystals were very hard and cubical (about 1 mm on an edge) and formed a honeycomb structure in the pipe. When all the sodium had been washed from the mass the mercury ran through the residual oxide matrix quite readily. The oxide adhered firmly to the walls in the first three sections but in others there was not enough oxide to bridge the pipe cross-section. After the mercury wash showed no alkalinity several more washes with mercury were performed to insure complete amalgamation. The residue was then dissolved in water, titrated with standard HCl and the solution kept for iron analyses. The mercury wash was titrated with standard HCl for total sodium amalgamated from each section. Upon completion of this analysis excess HCl was added to dissolve any iron residues but qualitative tests showed no iron in the mercury. Each section of pipe was etched on the inside with HCl and the washings analyzed separately for iron. The analytical results for each section are shown in the table below and plotted in Figure 2. (following page)

TABLE A						
Section No.	I	II	IIIA	IV *	VI	VII
gms Na	3.38	4.91	4.13		6.59	6.71
gms Na ₂ O	5.19	4.29	0.35		0.047	0.012
Vol. of pipe (cc)	7.15	7.15	4.98	7.15	6.74	7.15
Bulk density (gm Na ₂ O total gm)	0.605	0.466	0.078		0.0071	0.0018
Volume density (g Na ₂ O/cc)	0.715	0.600	0.070		0.0069	0.0017
Fe in residue (mg)	0.67	0.24	0.21	None	None	None
Fe on pipe wall (mg)	0.21	0.11	0.02	None	None	None
Total Fe (mg)	0.88	0.35	0.23	--	--	--

Sections IIIB and V could not be analyzed because these pipe sections would not fit the amalgamation chamber.

* This section was contaminated with oxygen during amalgamation; the iron concentration would not be affected and is shown.

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PART I FIGURE 2

Na₂O BULK DENSITY IN DRAIN LEG

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DISCUSSION OF RESULTS

Over the four month period of operation during which the loop was not drained, a total of 2.1 gms of oxygen as Na_2O_2 and 0.205 gms iron (from Armco iron corrosion specimens) had been added to the sodium via the sampling tank. The total oxide found in the drain leg was 2.54 gms as oxygen (exclusive of that in section IV and IIIB): this value is a little high because of contamination during sectioning and transfer and the possible introduction of oxygen to the system other than by Na_2O_2 additions. Considering the bulk densities presented in Table A, natural circulation cold traps could be expected to cold trap oxide until densities are attained such as those reached in the freeze seal. The 60% local bulk density reached in this case is significantly higher than the values calculated for the NaK and Na cold trapping experiments previously reported^(1, 2) (20% and 10% respectively). The evidence indicates that a cold trap could be designed which would cold trap to the local bulk densities found in the drain-fill pipe.

It is believed that most of the added iron had plated out throughout the loop, or possibly on the pipe surfaces nearest the iron sample for at no time did sodium samples show more than 20 ppm iron. The drain-fill pipe analyses show that iron is cold-trapped to some slight extent with the temperature of the main stream at 1000°F and the drain-fill pipe uninsulated (reaching room temperature at 12 - 13 inches from the hot stream). It is interesting to note also that iron was found in the drain-fill pipe oxide residue after amalgamation as well as on the drain-fill pipe walls.

The cold trapping effects of a freeze seal might be a serious deterrent to draining if too much oxide were allowed to collect there. The oxide probably would not completely plug the pipe but could seriously hinder flow upon draining. The crystal formation of the oxide presents a filtering section and it would become more plugged if liquid metal containing undissolved oxide crystals were drained through it. Therefore it would be best to drain liquid metals at temperatures above the saturation temperature of the oxide in the system. This technique should dissolve the oxide in the freeze seal. It might be well to note at this point that the practice of locally heating drain pipe to excessively high temperatures ($\sim 1400^\circ\text{F}$) might be detrimental to draining procedures since the oxide matrix may fuse and thereby cause a permanent plug.

This work is part of a continuing study of the behavior of oxygen and metallic impurities in sodium systems.

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Part II

The Effects of a Nickel Cold Trap on a Sodium-Filled Stainless Steel System

SUMMARY

This experiment showed that iron will move from a stainless steel system to a cold trap. The mechanism for the transfer of iron is postulated but the data needs substantiation before quantitative conclusions can be drawn.

INTRODUCTION

During previous studies of diffusion cold traps and the materials found in them after 3-5 months operation (1, 2) quantities of iron were always present in the cold trap residues. Since stainless steel cold traps had been used it was thought that a portion of the iron may have been contributed by oxide corrosion in the cold trap itself. In an effort to collect additional data on the migration of iron to cold traps, an all nickel trap was added to a stainless steel system containing circulating sodium at an oxide concentration of ≤ 0.005 wt. % oxygen.

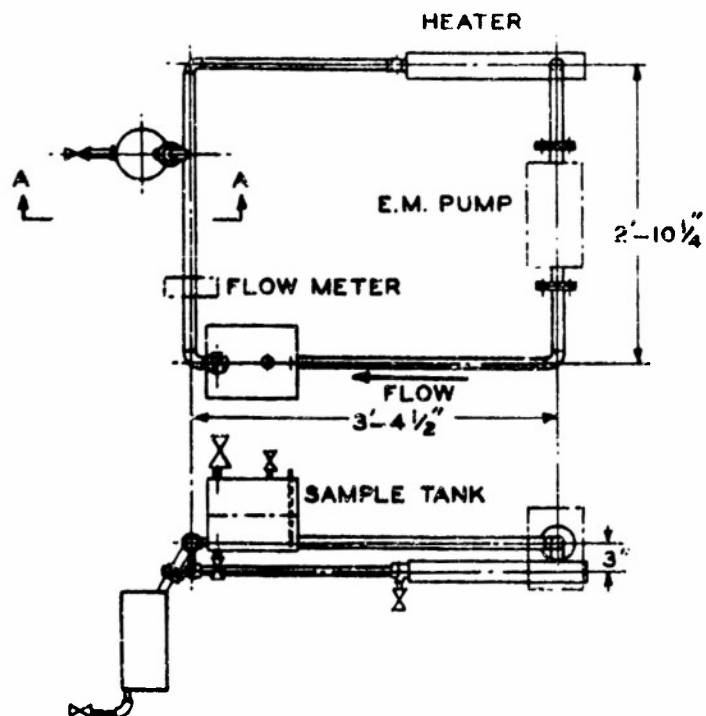
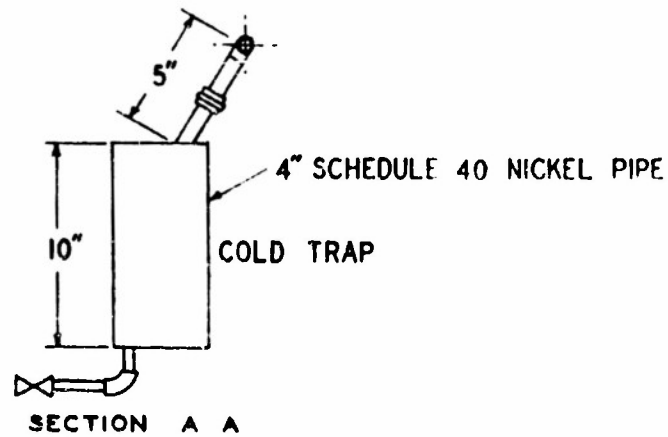
EXPERIMENTAL PROCEDURE

A cold trap was constructed of type A nickel (4 in. NPS pipe, 10 in. long) and attached by 5 in. of 1/2 in. pipe to a stainless steel system containing sodium at an oxygen concentration of ≤ 0.005 wt. %. See Part II, Figure 1 (following page) for a sketch of the system. The bottom of the cold trap was cooled with water circulating through a copper coil. Operating conditions were maintained as follows:

Sodium flow rate	8 gpm
Sodium temperature	1000°F (isothermal)
Starting O ₂ concentration	≤ 0.005 wt. %
Lowest temperature in cold trap	170 - 200°F

At the end of four months the sodium in the loop was allowed to freeze and the cold trap removed with its contents.

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NA LOOP & NATURAL CIRCULATION COLD TRAP
PART 2 FIGURE 1

C O N F I D E N T I A L

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ANALYTICAL PROCEDURE

The sodium in the cold trap was melted and drained at 250°F through a coarse sintered glass filter to trap any entrained particles of sodium oxide. During this entire procedure pure argon was kept on the system. Mercury was added to the cold trap to amalgamate the residual sodium and all additions were drained through the filter to trap oxide particles. It was assumed that the mercury washed all sodium from the filter and all residue was taken to be oxide only. All water and mercury washings were analyzed for iron; there was no iron in the mercury. The trap was washed with portions of 1:1 HCl until iron was no longer found in the washes. Iron samples were analyzed by a standard colorimetric method.

	Na ₂ O	Fe
On filter	2.15 g.	0.004 g.
Water wash of cold trap	1.26 g.	0.108 g.
1:1 HCl etch of cold trap	---	0.110 g.
Totals	3.14 g.	0.222 g.

Na₂O/Fe 15.4/1

DISCUSSION OF RESULTS

A correlation of data with prior cold trap results appears below.

Liquid Metal in System	NaK	Na	Na	Na
Type of Cold Trap	Stainless	Stainless	Nickel	Nickel
Total Run Time (months)	3	5	4	4
Total Na ₂ O in Cold Trap (gms)	154	86.1	3.41	See Table A Part I of this Report
Na ₂ O bulk density (wt. % Na ₂ O in cold trap contents)	21	10	0.2	
Gm Na ₂ O/cc of cold trap volume	0.19	0.11	0.003	
Total Fe in cold trap (gm)	2.40	0.76	0.22	
Ratio Na ₂ O/Fe	64:1	113:1	15:1	

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There are a number of factors entering into any comparisons made of the data above.

1. The amount of iron plated out on the walls of the stainless steel traps is unknown since only a water wash was used to get residual iron and oxide and acid etching would have been futile.
2. During the stainless cold trap testing the sodium (or NaK) had at times oxygen concentrations as high as 0.050 wt. %.
3. The cold traps were of two different materials; this may have a bearing on the behavior of iron.
4. The lowest temperature in the nickel trap was 170-200°F indicating a liquid solid interface: in the stainless traps there was only a liquid phase at 350-400°F.
5. The nickel trap was 4 in. pipe, 10 in. long and the stainless traps were 4 in. pipe, 4 in. long.

No definite quantitative conclusions on iron behavior can be drawn from the data above, but some observations can be made if the results are coupled with past experience in liquid metal work.

The experiments have proven one definite point - iron is moved from a stainless steel system to a cold trap. There is insufficient data to indicate how much iron can be expected to collect in cold traps as a function of time, cold trap size, geometry, etc. Since there was such a wide difference in system oxide concentrations for the stainless steel and nickel cold trap runs, it is not yet possible to indicate whether the iron was moved into the traps by temperature solubility differences alone or in conjunction with the movement of oxide.

An attractive possibility is to separate the iron found in the nickel cold trap as having two sources. The iron on the wall (etched off with acid) may be that which diffused into the trap as metallic iron; the remainder may have entered combined with the diffusing sodium monoxide. It was stated in Part I of this report that iron added to sodium is rapidly absorbed by the pipe surfaces in an all-nickel system. Therefore it might be expected that iron would diffuse more rapidly into the nickel cold trap on a stainless steel system because a rapid removal of iron to the nickel trap surface would create a higher concentration gradient for iron diffusion into the trap. In a stainless trap some of the iron concentration would consist

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of iron from the trap surface itself and thus buffer any iron diffusion from the primary system. The diffusion of metallic iron would not then be expected to be so rapid whereas the diffusion of iron in the form of an Na-Fe-O complex would be unaffected.

CONCLUSIONS

A nickel cold trap attached to a stainless system containing sodium with ≤ 0.005 wt. % oxygen will take small amounts of iron from the system. At higher oxygen levels more iron would be cold trapped.

It is believed some of the iron was moved to the nickel trap by temperature solubility differences and some by association with sodium monoxide. No quantitative data was obtained which could be applied to conclusive theories on the mechanism of iron transfer to cold traps.

Investigation of diffusion rates should be undertaken to elucidate the movement of metals in sodium and sodium-potassium alloys.

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